

**Imidazolines containing single-, twin- and triple-tailed hydrophobes
and hydrophilic pendants $(\text{CH}_2\text{CH}_2\text{NH})_n\text{H}$ as inhibitors of mild steel
corrosion in CO_2 -0.5 M NaCl**

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Supplementary Materials

2. Experimental

2.3. Synthesis

2.3.1.1. General procedure for the synthesis of di-, and tri-octyloxybenzamides (3b, 3c). A mixture of alkoxybenzoic acid (**2b** or **2c**) (60 mmol) in SOCl_2 (17 cm^3) was heated to 80 °C for 30 min. After removal of the excess SOCl_2 , the residual liquid was added drop wise to a 30% NH_3 solution (150 cm^3) at 0 °C. The benzamide (**3b** or **3c**) was filtered and dried.

2.3.1.1. 3, 5-Dioctyloxybenzamide (3b). Yield: 88%; Mp 85-86°C. (Found: C, 73.0; H, 10.2; N, 3.6. $\text{C}_{23}\text{H}_{39}\text{NO}_3$ requires C, 73.17; H, 10.41; N, 3.71%). ν_{max} (KBr) 3383, 3203, 2923, 2854, 1649, 1597, 1466, 1435, 1389, 1254, 1169, 1119, 1058, 847, and 682 cm^{-1} . δ_{H} (CDCl_3) 0.88 (6H, t, J 7.0 Hz), 1.30 (16H, m), 1.43 (4H, m), 1.76 (4H, m), 3.96 (4H, t, J 6.7 Hz), 6.10 (2H, br), 6.59 (1H, s), 6.91 (2H, s). δ_{C} (CDCl_3 , 45 °C): 14.08 (2C), 22.66 (2C), 25.98 (2C), 29.15 (2C), 29.21 (2C), 29.30 (2C), 31.78 (2C), 68.32 (2C), 104.87, 105.63 (2C), 135.30, 160.37 (2C), 169.51.

2.3.1.2. 3, 4, 5-Trioctyloxybenzamide (3c). Yield: 90%; Mp 84-85 °C. (Found: C, 73.4; H, 10.8; N, 2.7. $\text{C}_{31}\text{H}_{55}\text{NO}_4$ requires C, 73.62; H, 10.96; N, 2.77%). ν_{max} (KBr) 3358, 3192, 2921, 2851, 1648, 1580, 1465, 1428, 1382, 1317, 1237, 1122, 1017, 852, and 691 cm^{-1} . δ_{H} (CDCl_3) 0.88 (9H, t, J 7.0 Hz), 1.30 (24H, m), 1.45 (6H, m), 1.74 (2H, m), 1.81 (4H, m), 4.00 (6H, t, J 6.7 Hz), 6.00 (2H, br), 7.01 (2H, s). δ_{C} (CDCl_3): 14.08 (3C), 22.65 (3C), 26.03 (3C),

29.26 (3C), 29.32 (3C), 29.49 (2C), 30.28, 31.80 (2C), 31.87, 69.31 (2C), 73.49, 106.04 (2C), 128.14, 141.51, 153.02 (2C), 169.37.

2.3.2. General procedure for the synthesis of di-, and tri-alkoxybenzonnitriles (4b, 4c). A mixture of octyloxybenzamide (**3b** or **3c**) (45 mmol) in SOCl_2 (70 mmol) in benzene (20 cm^3) was heated to 80 °C for 1 h or until the reaction was complete as indicated by TLC experiment (silica, $\text{Et}_2\text{O}/\text{MeOH}$ 9:1). After removal of the excess SOCl_2 , the residual liquid was crystallized from pentane to give the benzonitrile (**4b** or **4c**).

2.3.2.1. 3, 5-Dioctyloxybenzonitrile (4b). Yield: 85%; (Found: C, 76.5; H, 10.5; N, 3.8. $\text{C}_{23}\text{H}_{37}\text{NO}_2$ requires C, 76.83; H, 10.37; N, 3.90%). ν_{max} (KBr) 2926, 2854, 2230, 1590, 1442, 1386, 1349, 1299, 1255, 1173, 1056, 845, 723, and 677 cm^{-1} . δ_{H} (CDCl_3) 0.89 (6H, t, J 7.0 Hz), 1.30 (16H, m), 1.43 (4H, m), 1.77 (4H, m), 3.93 (4H, t, J 6.7 Hz), 6.63 (1H,s), 6.73 (2H, s). δ_{C} (CDCl_3): 14.07 (2C), 22.63 (2C), 25.92 (2C), 28.99 (2C), 29.18 (2C), 29.25 (2C), 31.77 (2C), 68.82 (2C), 106.45, 110.19 (2C), 113.18, 118.90, 160.44 (2C).

2.3.2.2. 3,4,5-Trioctyloxybenzonitrile (4c). Yield: 87%; Mp 39-40 °C; (Found: C, 76.1; H, 10.8; N, 2.7. $\text{C}_{31}\text{H}_{53}\text{NO}_3$ requires C, 76.34; H, 10.95; N, 2.87%). ν_{max} (KBr) 2922, 2852, 2225, 1581, 1502, 1467, 1428, 1386, 1339, 1240, 1129, 1019, 977, 837, 815, 723, and 624 cm^{-1} . δ_{H} (CDCl_3) 0.89 (9H, overlapping t, J 7.0 Hz), 1.31 (24H, m), 1.45 (6H, m), 1.73 (2H, m), 1.80 (4H, m), 3.96 (4H, t, J 6.7 Hz), 4.00 (2H, t, J 6.5 Hz), 6.81 (2H,s). δ_{C} (CDCl_3): 14.11 (3C), 22.68

(3C), 26.02 (3C), 29.17 (2C), 29.27 (3C), 29.31 (2C), 29.48, 30.29, 31.82 (2C), 31.90, 69.40 (2C), 73.72, 106.17, 110.45 (2C), 118.90, 142.30, 153.46 (2C).

2.3.3.1. 1-(2-Aminoethyl)-2-(3,5-dioctyloxyphenyl)-2-imidazoline (7b). Yield: 87%; ν_{\max} . (neat) 3450, 3283, 2925, 2855, 1585, 1438, 1408, 1386, 1344, 1287, 1167, 1056, 1003, 837, and 735 cm^{-1} . δ_{H} (CDCl_3): 0.89 (6H, t, J 6.7 Hz), 1.20-1.50 (22 H, m), 1.76 (4H, m), 2.84 (2H, t, J 6.1 Hz), 3.13 (2H, t, J 6.1 Hz), 3.43 (2H, t, J 9.8 Hz), 3.89 (2H, t, J 9.8 Hz), 3.96 (4H, t, J 6.4 Hz), 6.49 (1H, s), 7.31 (2H, s). δ_{C} (CDCl_3): 13.96 (2C), 22.51 (2C), 25.37 (2C), 29.05 (2C), 29.09 (2C), 29.18 (2C), 31.66 (2C), 40.87, 51.21, 52.49, 53.30, 68.07 (2C), 102.97, 106.39 (2C) 133.03, 160.05 (2C), 168.01.

2.3.3.2. 1-(2-Aminoethyl)-2-(3,4,5-trioctyloxyphenyl)-2-imidazoline (7c). Yield: 95%; ν_{\max} . (neat) 3290, 2926, 2854, 1577, 1503, 1466, 1425, 1380, 1340, 1234, 1114, 1006, 841 and 722 cm^{-1} . δ_{H} (CDCl_3): 0.89 (9H, t, J 6.7 Hz), 1.20-1.50 (32 H, m), 1.72 (2H, m), 1.79 (4H, m), 2.86 (2H, t, J 6.1 Hz), 3.15 (2H, t, J 6.1 Hz), 3.45 (2H, t, J 9.8 Hz), 3.89 (2H, t, J 9.8 Hz), 3.96 (6H, m), 6.77 (2H, s). δ_{C} (CDCl_3): 14.12 (3C), 22.08 (3C), 26.08 (3C), 29.30 (5C), 29.36 (2C), 29.54, 30.29, 31.18 (2C), 31.91, 41.09, 51.57, 52.93, 53.41, 69.19 (2C), 73.44, 106.78 (2C), 126.17, 139.37, 153.02 (2C), 168.30.

2.3.4.1. 1-[2-{2-(2-Aminoethylamino)ethylamino}ethyl] -2-(3, 5-dioctyloxyphenyl)-2-imidazoline (8b). Yield: 88%. ν_{\max} . (neat) 3287, 2926, 2855, 1586, 1459, 1439, 1385, 1344, 1164, 1052, 835, and 724 cm^{-1} . δ_{H} (CDCl_3): 0.89 (6H, t, J 6.7 Hz), 1.20-1.65 (24H, m), 1.75 (4H,

m), 2.68 (2H, t, J 5.7 Hz), 2.74 (4H, s), 2.78 (2H, t, J 6.5 Hz), 2.81 (2H, t, J 6.1 Hz), 3.19 (2H, t, J 6.4 Hz), 3.42 (2H, t, J 9.7 Hz), 3.93 (2H, t, J 9.7 Hz), 3.97 (4H, t, J 6.4 Hz), 6.48 (1H, s), 6.66 (2H, s). δ_C (CDCl₃): 14.11 (2C), 22.66 (2C), 26.02 (2C), 29.24 (2C), 29.33 (2C), 31.38 (2C), 31.82 (2C), 41.77, 48.50, 49.30, 49.62, 49.81, 51.50, 52.51, 53.41, 68.20 (2C), 103.12, 106.55 (2C), 133.11, 160.17 (2C), 168.07.

2.3.4.2. 1-[2-{2-(2-Aminoethylamino)ethylamino}ethyl] -2-(3,4,5-trioctyloxyphenyl)-2-imidazoline (8c). Yield: 92%. ν_{\max} . (neat) 3287, 2926, 2855, 1576, 1501, 1466, 1426, 1379, 1341, 1234, 1114, 1007, 957, 843, and 722 cm⁻¹. δ_H (CDCl₃): 0.89 (9H, t, J 6.7 Hz), 1.20-1.65 (34H, m), 1.75 (6H, m), 2.66 (2H, t, J 5.8 Hz), 2.72 (4H, s), 2.77 (2H, t, J 6.7 Hz), 2.79 (2H, t, J 6.0 Hz), 3.21 (2H, t, J 6.4 Hz), 3.45 (2H, t, J 9.7 Hz), 3.88 (2H, t, J 9.7 Hz), 3.97 (6H, t, J 6.4 Hz), 6.78 (2H, s). δ_C (CDCl₃): 14.12 (3C), 22.68 (2C), 22.70, 26.04 (3C), 29.30 (3C), 29.36 (2C), 29.38 (2C), 29.55, 30.30, 31.84 (2C), 31.91, 41.80, 48.62, 49.35, 49.66, 50.09, 51.66, 52.53, 53.39, 69.12 (2C), 73.39, 106.75 (2C), 126.18, 139.33, 152.97 (2C), 168.16.